

# PATENT COOPERATION TREATY

## PCT

### INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference <b>85 DP 01 L49</b>	<div style="display: flex; justify-content: space-between;"> <div> <b>FOR FURTHER ACTION</b> </div> <div>           See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)         </div> </div>	
International application No. <b>PCT/GB98/03056</b>	International filing date (day/month/year) <b>09/10/1998</b>	Priority date (day/month/year) <b>10/10/1997</b>
International Patent Classification (IPC) or national classification and IPC <b>D06M13/00</b>		
Applicant <b>E.I. DUPONT DE NEMOURS AND COMPANY et al.</b>		
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 9 sheets, including this cover sheet.</p> <p><input checked="" type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of 4 sheets.</p>		
<p>3. This report contains indications relating to the following items:</p> <ul style="list-style-type: none"> <li>I <input checked="" type="checkbox"/> Basis of the report</li> <li>II <input type="checkbox"/> Priority</li> <li>III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</li> <li>IV <input type="checkbox"/> Lack of unity of invention</li> <li>V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</li> <li>VI <input type="checkbox"/> Certain documents cited</li> <li>VII <input checked="" type="checkbox"/> Certain defects in the international application</li> <li>VIII <input checked="" type="checkbox"/> Certain observations on the international application</li> </ul>		
Date of submission of the demand  <b>16/03/1999</b>	Date of completion of this report  <div style="text-align: right; font-size: 1.2em;"><b>0 5. 01. 00</b></div>	
Name and mailing address of the international preliminary examining authority:  <div style="display: flex; align-items: center;"> <div>             European Patent Office              D-80298 Munich              Tel. +49 89 2399 - 0 Tx: 523656 epmu d              Fax: +49 89 2399 - 4465           </div> </div>	Authorized officer  <b>Santavicca, G</b>  Telephone No. +49 89 2399 8632	



**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/GB98/03056

**I. Basis of the report**

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

**Description, pages:**

1-32 as originally filed

**Claims, No.:**

1-11 as received on 18/11/1999 with letter of 18/11/1999

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:  
☒ the claims, Nos.: 11, 14 as initially filed  
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

**1. Statement**

Novelty (N)	Yes:	Claims 1-11
	No:	Claims
Inventive step (IS)	Yes:	Claims
	No:	Claims 1-11 (see Section V)
Industrial applicability (IA)	Yes:	Claims 1-11
	No:	Claims

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**2. Citations and explanations**

**see separate sheet**

**VII. Certain defects in the international application**

The following defects in the form or contents of the international application have been noted:

**see separate sheet**

**VIII. Certain observations on the international application**

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

**see separate sheet**

## NOTES TO FORM PCT/ISA/220

These Notes are intended to give the basic instructions concerning the filing of amendments under article 19. The Notes are based on the requirements of the Patent Cooperation Treaty, the Regulations and the Administrative Instructions under that Treaty. In case of discrepancy between these Notes and those requirements, the latter are applicable. For more detailed information, see also the PCT Applicant's Guide, a publication of WIPO.

In these Notes, "Article", "Rule", and "Section" refer to the provisions of the PCT, the PCT Regulations and the PCT Administrative Instructions respectively.

### INSTRUCTIONS CONCERNING AMENDMENTS UNDER ARTICLE 19

The applicant has, after having received the international search report, one opportunity to amend the claims of the international application. It should however be emphasized that, since all parts of the international application (claims, description and drawings) may be amended during the international preliminary examination procedure, there is usually no need to file amendments of the claims under Article 19 except where, e.g. the applicant wants the latter to be published for the purposes of provisional protection or has another reason for amending the claims before international publication. Furthermore, it should be emphasized that provisional protection is available in some States only.

#### What parts of the international application may be amended?

Under Article 19, only the claims may be amended.

During the international phase, the claims may also be amended (or further amended) under Article 34 before the International Preliminary Examining Authority. The description and drawings may only be amended under Article 34 before the International Examining Authority.

Upon entry into the national phase, all parts of the international application may be amended under Article 28 or, where applicable, Article 41.

#### When?

Within 2 months from the date of transmittal of the international search report or 16 months from the priority date, whichever time limit expires later. It should be noted, however, that the amendments will be considered as having been received on time if they are received by the International Bureau after the expiration of the applicable time limit but before the completion of the technical preparations for international publication (Rule 46.1).

#### Where not to file the amendments?

The amendments may only be filed with the International Bureau and not with the receiving Office or the International Searching Authority (Rule 46.2).

Where a demand for international preliminary examination has been/is filed, see below.

#### How?

Either by cancelling one or more entire claims, by adding one or more new claims or by amending the text of one or more of the claims as filed.

A replacement sheet must be submitted for each sheet of the claims which, on account of an amendment or amendments, differs from the sheet originally filed.

All the claims appearing on a replacement sheet must be numbered in Arabic numerals. Where a claim is cancelled, no renumbering of the other claims is required. In all cases where claims are renumbered, they must be renumbered consecutively (Administrative Instructions, Section 205(b)).

The amendments must be made in the language in which the international application is to be published.

#### What documents must/may accompany the amendments?

##### Letter (Section 205(b)):

The amendments must be submitted with a letter.

The letter will not be published with the international application and the amended claims. It should not be confused with the "Statement under Article 19(1)" (see below, under "Statement under Article 19(1)").

The letter must be in English or French, at the choice of the applicant. However, if the language of the international application is English, the letter must be in English; if the language of the international application is French, the letter must be in French.

## NOTES TO FORM PCT/ISA/220 (continued)

The letter must indicate the differences between the claims as filed and the claims as amended. It must, in particular, indicate, in connection with each claim appearing in the international application (it being understood that identical indications concerning several claims may be grouped), whether

- (i) the claim is unchanged;
- (ii) the claim is cancelled;
- (iii) the claim is new;
- (iv) the claim replaces one or more claims as filed;
- (v) the claim is the result of the division of a claim as filed.

The following examples illustrate the manner in which amendments must be explained in the accompanying letter:

1. [Where originally there were 48 claims and after amendment of some claims there are 51]:  
"Claims 1 to 29, 31, 32, 34, 35, 37 to 48 replaced by amended claims bearing the same numbers; claims 30, 33 and 36 unchanged; new claims 49 to 51 added."
2. [Where originally there were 15 claims and after amendment of all claims there are 11]:  
"Claims 1 to 15 replaced by amended claims 1 to 11."
3. [Where originally there were 14 claims and the amendments consist in cancelling some claims and in adding new claims]:  
"Claims 1 to 6 and 14 unchanged; claims 7 to 13 cancelled; new claims 15, 16 and 17 added." or  
"Claims 7 to 13 cancelled; new claims 15, 16 and 17 added; all other claims unchanged."
4. [Where various kinds of amendments are made]:  
"Claims 1-10 unchanged; claims 11 to 13, 18 and 19 cancelled; claims 14, 15 and 16 replaced by amended claim 14; claim 17 subdivided into amended claims 15, 16 and 17; new claims 20 and 21 added."

### "Statement under article 19(1)" (Rule 46.4)

The amendments may be accompanied by a statement explaining the amendments and indicating any impact that such amendments might have on the description and the drawings (which cannot be amended under Article 19(1)).

The statement will be published with the international application and the amended claims.

It must be in the language in which the international application is to be published.

It must be brief, not exceeding 500 words if in English or if translated into English.

It should not be confused with and does not replace the letter indicating the differences between the claims as filed and as amended. It must be filed on a separate sheet and must be identified as such by a heading, preferably by using the words "Statement under Article 19(1)."

It may not contain any disparaging comments on the international search report or the relevance of citations contained in that report. Reference to citations, relevant to a given claim, contained in the international search report may be made only in connection with an amendment of that claim.

### Consequence if a demand for international preliminary examination has already been filed

If, at the time of filing any amendments under Article 19, a demand for international preliminary examination has already been submitted, the applicant must preferably, at the same time of filing the amendments with the International Bureau, also file a copy of such amendments with the International Preliminary Examining Authority (see Rule 62.2(a), first sentence).

### Consequence with regard to translation of the international application for entry into the national phase

The applicant's attention is drawn to the fact that, where upon entry into the national phase, a translation of the claims as amended under Article 19 may have to be furnished to the designated/elected Offices, instead of, or in addition to, the translation of the claims as filed.

For further details on the requirements of each designated/elected Office, see Volume II of the PCT Applicant's Guide.

**Re Item V**

**Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

- 0.1 The claims have been amended by incorporation of the subject matter of claim 4 as initially on file into the amended claims 3, 8, 10 and 11, whereby the modification is allowable as these amendments do not go beyond the disclosure in the international application as filed.
- 0.2 By incorporation of the subject matter of claim 4 as initially on file, the subject matter claimed by the independent claims is limited to such methods, textiles and fibers which all use or comprise fragrance compositions as defined in claim 4 as initially on file. The disclosure of original claim 4 defines fragrance materials (categories A and B) by the possible classes of compounds and in addition by two physical properties (octanol/water partition coefficient, gas chromatographic Kovats index) which values must lie above specific limits. The determination of the physical values as defined in claim 4 as originally on file can be achieved by standard techniques as is e.g. disclosed on page 8, lines 19 to page 9, line 21.
- 0.3 Moreover, numerous examples for applicable components falling within category A (resp. sub-category A') or category B (resp. sub-category B') are disclosed in the specification (page 12, line 5 to page 13, line 19; page 13, line 14 to 18; page 14, line 21 to page 17, line 19; page 17, line 24 to page 18, line 14).
- 0.4 Therefore, a person skilled in the art is able to carry out the present invention without undue burden within substantially the whole scope of the amended claims. This is true not only for the substance claims but also for the method claims.
1. Before assessing the novelty and the inventiveness of the claimed subject-matter, it is necessary to consider the wording of claims 1, 8, 10 and 11 - Art. 6 PCT as to clarity and interpretation of claims - then the pertinence of the cited documents.
- 1.1 Claim 1 concerns any method (i.e. any activity of) treating (any treatment, e.g. washing) any textile which is (in form of) yarn (i.e. not yet made into a garment) or

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unworn fabric (e.g. any new fabric which has been bought but not yet been worn) containing (ie including together with other fibres any amount of) spandex fibres (see the description for the definition of these fibres). The method comprises (i.e. includes the features as listed in the body of the claim but does not exclude that other features be present) contacting (in any way, e.g. by washing) the textile (why not the yarn too?) with (any amount of) a(ny) perfume composition (i.e. any substance having a pleasant smell, e.g. as contained in many detergents) which perfume (normally) is a mixture of (any amount of any kind of) fragrance materials (i.e. of materials having pleasant influence), so that fragrance materials are deposited on the textile (not necessarily also retained, nor necessarily deposited and retained at a greater concentration on the spandex fibres than on the other fibres). The perfume composition contains (ie includes among other materials) at least 50%, by weight of the perfume composition, of fragrance materials selected from the categories A and B as defined. To note that both the common logarithms and the Kovats index may be any higher than the minimum values as defined.

- 1.2 Claims 8, 10 and 11 all concern any textile (claims 8 and 10) and spandex fibre (claim 11) with any amount of the fragrance material deposited as defined, whereby these products do not necessarily result from the methods of claims 1 and 3 respectively. It is worth mentioning that the products as defined in claims 8, 10 and 11 actually may carry any amount of fragrance as deposited thereon, as a case in point even much less than 0.001%, ie much less than the minimum
- 2.1 According to the description, the fragrance materials of categories A and B, or A' and B', actually are able to deposit and then be retained better on spandex fibres than on a number of other textile fibres (see page 3, first paragraph). These (number of) deposited fragrances will still be perceptible on the fabric, even after several washes of the so treated fabrics using laundry products with a different perfume (see page 4, lines 1-3). The perfume composition of the present, alleged invention concentrates preferably on spandex fibres (see page 4, lines 7-9). This is not reflected in the claims, apart from claim 10, which is distinguished over claim 8 by this feature, which feature however should be common to all of the claims.

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- 2.2 The products as defined in claims 8, 10 and 11, do not necessarily result from the methods of claims 1 and 3, and do not necessarily include the amounts as defined in the additional features of claims 2 and 9.
- 2.3 It is apparent that the independent claims do not include all of the essential features of the alleged invention, i.e. those features (like the preferential deposition and retention on spandex fibres as well as the amounts deposited on the claimed products) which are necessary to solve the technical problem of the alleged invention, or which are presented as necessary to achieve the advantages used in the description to justify the presence of an inventive step. Hence, the requirements of Art. 6 PCT as to support by the description are not met.
- 2.4 Any question of support taken apart, if the products of any of claims 8, 10 and 11 do not result from the methods of claims 1 and 3 with the additional features of claim 2, and do not contain either enough amount of fragrance/perfume as given in the additional features of claim 9, ie they do not include all of the essential features of the alleged invention, they thus encompass embodiments of the (alleged) invention which are not suitable for achieving the object or the advantages of the alleged invention as described. In other words, there is a lack of support for the presence of an inventive step "a priori", i.e. before considering the relevance of the prior art (Art. 33(3) PCT).
3. Pertinence of the available art
- D1 (**GB 1596792A**) discloses a fabric conditioning composition comprising an effective amount of a fabric softener and a deodorizing amount of a deodorant perfume (claim 1). The deodorant perfume according to D1 has to comprise materials which depress the partial vapor pressure of morpholine but actually includes fragrances A and B (see the formulation of example 1 - iso-eugenol; Patchouli oil - p-t-butylcyclohexyl acetate; 2-n-heptylcyclopentanone;  $\alpha$ -iso-methyl ionone;  $\gamma$ -undecalactone - as well as formulations C1 and D2 of examples 4-7). These formulations actually are deposited on polyester fabrics and spandex fibers are not mentioned in D1. As a fabric softener is not part of the present application, the treatment concerns spandex fibers and the claimed fragrance materials are preferentially deposited on spandex fibers (this is not yet defined in claim 1 but should actually result as an effect) than on the other fibers, the subject matter of



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the present applicaion is novel over D1. Moreover, as D1 gives no hint to use the given, specific selection of perfume materials for textiles containing spandex fibers or for spandex fibers as is disclosed in the present application for achieving the inventive advantages, the subject matter of the present application has to be considered not only as novel but also as inventive in respect to D1.

D2 (DE 296 00 628 U1) describes a vaporization liquid for steam irons, which may comprise a perfume compound, which may include inter alia (A) **citronellol** and (B) **citronellal**, see page 3, penultimate line, and claim 4. Therefore, this document discloses one of the numerous methods by which a fragrance material can be applied to a textile. However, no hint can be found to use fragrance materials as are disclosed in the present application on spandex fibers. Therefore the subject matter of the present application is novel and inventive over D2.

D3 (DE 40 11 928 A1) discloses a textile (inter alia "dessous", which may contain Spandex fibers, but this is not disclosed actually) with an impregnation consisting of fragrance materials (not specifically disclosed, nor exemplified either) together with additional means to make said fragrance materials sustainable and resistant to cleaning agents. Said additional means are either waxes or wax-like components or substances in which the fragrance materials have a good solubility. Finally, D3 discloses micro capsules as additional means. From that it becomes evident that D3 has a quite different teaching as compared to the present application and, therefore, the subject matter of the present application is novel and inventive in respect to D3 too.

D4 (DE 4 242 327 A1) discloses a method for finishing textile materials such as panty hose, which may include spandex fibers (this is however not disclosed) by applying micro capsules into the knit fabric. One of the possible contents of the micro capsules are perfume materials, whose composition is not disclosed. Hence, no hint can be found to chose one of the fragrance materials as disclosed in the present application to achieve the advantageous deposit on spandex fibers. Therefore, also D4 does not disclose any subject matter which can destroy novelty and inventive step of the subject matter in the present application.

D5 (EP 0 581 274 A1) discloses a method comprising the steps of cationising a textile product with a liquid containing a nitrogenous cationic compound and fixing micro-capsules which are made essentially of a polymer compound and which contain a perfume to the textile product. According to the examples, see page 5,

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lines 18-33, the perfume composition may include fragrances A and B as defined in the claims of the present application. As a case in point: (A) citronellol, geraniol, linalool, eugenol, menthol, terpineol, isoamyl salicylate; (B) ionone and methylionone,  $\alpha$ -amylcinnamaldehyde, methyl methylanthranilate, jasmone and  $\gamma$ -undecalactone. The fibers which may be treated are defined on page 3, lines 8-11. Spandex fibers are not mentioned however. Therefore, D5 cannot anticipate either the subject-matter as defined in the present application.

D6 (WO-A-93/14259) discloses a method for applying substances to fibre materials and textile substrates from a liquid phase, in which a supercritical fluid or a liquefied gas or gas mixture is used as a fluid. No hint can be found in the description of D6 to use fragrance materials as they are used in the present invention on spandex fibers.

D7 (JP 1272875 A) concerns a fragrant non-woven fabric with elastic fibers (spandex not disclosed) on which micro capsules are adhered.

D8 (JP 6146112 A) concerns a two layer spandex fibre in which one layer comprises a Ag based inorganic antibacterial agent to give the two layer spandex fibre antibacterial properties. Fragrances A and B are not disclosed.

D9 (EP 0 841391 A1) ( see particularly page 12, line 33, to page 17, line 40) discloses a hydrophilic perfume composition comprising a perfume with fragrances A and B (see the compounds as listed in the tables), whereby the ingredients (fragrances) have at least partly a Clog P of 3 or smaller, and an amino functional polymer. No disclosure or hint is given to use said perfume composition on spandex fibers.

Therefore, also these three last documents D7-D9 do neither disclose the subject matter of the present application, nor do they render the use of the fragrance composition on spandex fibers according to the present application obvious.

4. To sum up, the subject matter of the present application, if amended to overcome the objections as raised, would appear to fulfil the requirements of Art. 33 PCT, as neither the "general background knowledge", nor the above discussed documents disclose the subject matter of the present application, and, further, neither alone nor in combination the documents forming the state of the art make it obvious to use the fragrance compositions on spandex fibers as they are used in the subject matter of the present application to achieve the disclosed advantages.

**Re Item VII**

**Certain defects in the international application**

0. The following objections/observations are merely for the sake of information, i.e. to be used before the national or regional further instance, if any.
1. The documents cited in the search report do not reflect any close state of the art and need not be acknowledged in the description - Rule 5.1(a)(ii) PCT, apart perhaps from the documents disclosing fragrances A and B on other fibres, and particularly D9, which discloses the parameters used in the present invention. The comment, if any, however should be purely factual - Art. 34.2(b) PCT.
2. The description should be brought into line with any new set of claims - Art. 6 PCT.
3. To expedite national or regional, further examination, if any, the applicant(s) should indicate the location in the application as originally filed of the passages forming a basis for the amendments - Art. 34.2(b) PCT.

**Re Item VIII**

**Certain observations on the international application**

1. The presence of two independent method claims (see claims 1 and 3) and of three independent product claims (see particularly claims 8 and 10, but also 11) is such that the entirety of the claims is not concise as requested by Art. 6 PCT. In the present case, it is apparent that one independent claim in any category actually is appropriate. The only one independent claim in any category might be drawn in the form with alternatives, namely "of treating a spandex fibre or a textile which is yarn or unworn fabric containing spandex fibres, comprising ...".

## PATENT COOPERATION TREATY

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ALLEMAGNE	
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05.01.00

Applicant's or agent's file reference  
85 DP 01 L49

IMPORTANT NOTIFICATION

International application No.  
PCT/GB98/03056International filing date (day/month/year)  
09/10/1998Priority date (day/month/year)  
10/10/1997

Applicant

E.I. DUPONT DE NEMOURS AND COMPANY et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

## 4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification <sup>6</sup> :</b> <b>D06M 13/00</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 99/19553</b> <b>(43) International Publication Date:</b> 22 April 1999 (22.04.99)
<b>(21) International Application Number:</b> PCT/GB98/03056 <b>(22) International Filing Date:</b> 9 October 1998 (09.10.98)  <b>(30) Priority Data:</b> 9721588.3 10 October 1997 (10.10.97) GB  <b>(71) Applicant (for all designated States except US):</b> E.I. DUPONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US).  <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> PERRING, Keith, Douglas [GB/GB]; 14 Malvern Road, Ashford, Kent TN24 8HS (GB). CLEMENTS, Christopher, Francis [GB/GB]; Inveresk, Lookers Lane, Saltwood, Hythe, Kent CT21 5HW (GB). MACMASTER, Angus, Peter [GB/GB]; The Barn, Robus Ceramics, Evington Park, Hastingleigh, Nr Ashford, Kent TN25 5JH (GB). PALMEN, Raymond [NL/CH]; 3, chemin de Passoret, CH-1234 Vessy (CH). JOHN, Olivier, William [CH/CH]; 7, rue Monnier, CH-1207 Geneva (CH).  <b>(74) Agents:</b> KUHNEN, Rainer, A. et al.; Alois-Steinecker-Strasse 22, D-85354 Freising (DE).		<b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> METHOD FOR TEXTILE TREATMENT FOR SPANDEX CONTAINING FABRICS  <b>(57) Abstract</b>  A method of treating textile which is yarn or fabric containing spandex fibres, comprising contacting the textile with a perfume so that the perfume is deposited on the fabric. The perfume contains a mixture of fragrance materials which preferentially deposit on the spandex fibres. The yarn of fabric may be made up into garments.		

**FOR THE PURPOSES OF INFORMATION ONLY**

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CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	NZ	New Zealand		
CM	Cameroon			PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	SG	Singapore		

## METHOD FOR TEXTILE TREATMENT FOR SPANDEX CONTAINING FABRICS

This invention relates to the treatment of textiles containing spandex fibres.

At the present time, many garments are made  
5 from fabric which contains a mixture of fibres, a proportion of which are elastic, so that the fabric has the ability to stretch and to recover from stretch. Spandex fibres are commonly used for this purpose. The term "Spandex" has been adopted as a  
10 generic term by the United States Federal Trade Commission to denote a manufactured fibre in which the fibre-forming substance is a long chain synthetic polymer composed of at least 85% of a segmented polyurethane. A discussion of such fibres can be found  
15 in "History of Spandex Elastomeric Fibres" by A.J. Ultee, which is a chapter starting at page 278 in *Man-Made Fibres: Their Origin and Development*, edited by R.V. Seymour and R.S. Porter, Elsevier 1993. Spandex fibres are also referred to as "elastane" or  
20 "elasthane" fibres.

Another discussion of such fibres is found under the heading "Segmented Polyurethanes" at page 613 of *Handbook of Textile Fibres* by J. Gordon Cook, 5th Ed. Merrow Publishing Company 1984. Further

description of elastanes and their applications can be found in "Synthesefasern: Grundlagen, Technologie, Verarbeitung und Anwendung", B von Falkei (editor), Verlag Chemie (1981). Commercially available elastanes  
5 are well known, in particular as sold under the name LYCRA®, a registered trade mark of DuPont de Nemours and Company. Patents relating to such fibres include US-A-5000899, US-A-5288779 and US-A-5362432.

The deposition of perfume onto garments and  
10 other fabrics during laundering has been established for many years. Perfume is incorporated into laundry products such as detergent compositions for fabric washing and rinse conditioners for softening the fabrics.

15 Although the perfume serves to cover the base odour of such a product and to give the unused product an attractive fragrance, it also deposits on the fabric.

Certain perfumes have the ability to provide  
20 deodorant action against body odour, either when directly applied to human skin, or when included in a laundry product. Such perfumes are described in EP-B-3172, US-A-4304679, US-A-4278658, US-A-4134838, US-A-4288341 and US-A-4289641, US-A-5482635 and US-A-  
25 5554588.



Summary of the Invention

We have now found that a number of fragrance materials used in perfumery are able to deposit and then be retained better on spandex fibres than on a  
5 number of other textile fibres.

Delivery of fragrance materials to fabric can take place during washing, as is well known. The present invention appreciates that the application of perfume to textiles containing spandex fibres can be  
10 utilised in the treatment of textiles which are newly made - that is to say textile goods which have never been worn as garments by a consumer.

Therefore in one aspect the present invention provides a method of treating textile which  
15 is yarn or fabric containing spandex fibres, comprising contacting the textile with a perfume so that the perfume is deposited on the fabric. Preferably, the fabric is unworn. It may have been made up into a garment.

20 In a related second aspect, the invention provides textile which is yarn or fabric containing spandex fibres, having perfume deposited on the textile. Preferably, the fabric is unworn. It may have been made up into a garment.

25 We have observed that a range of fragrance materials deposited on such textiles will still be

perceptible on the fabric, even after several washes of the fabric using laundry products with a different perfume, or none.

The invention also provides use of a perfume composition in the treatment of textile which is yarn or fabric containing spandex and other fibres, to deposit fragrance materials at a greater concentration on the spandex fibres than on the other fibres. Preferably, the fabric is unworn.

In significant forms of this invention, the perfume used to treat the textile (or the combination of fragrance materials deposited thereon) is a deodorant perfume. Then when the textile is made into a garment, that garment will have an in-built deodorant property.

#### Detailed description

The various aspects of this invention, preferred forms and materials useful therein will now be discussed in greater detail.

#### 20 Textiles

The textiles to which this invention relates include spandex fibres. As mentioned earlier, this term denotes a manufactured fibre in which the fibre forming substance is a long chain synthetic polymer compound composed of at least 85% of a segmented polyurethane.

Thus the polymer which is spun into spandex fibres is a copolymer incorporating urethane linkages. Generally the polymer contains so-called soft (i.e. lower melting) segments which may be polyalkylene ethers or polyesters and so-called hard (i.e. higher melting) segments which are portions derived from the reaction of an isocyanate and a chain extender which is typically a diamine.

The soft segments may be poly(tetramethylene)ethers, possibly containing substituted tetramethylene glycol residues as described in US-A-5000899. Organic diisocyanates which may be used include conventional diisocyanates, such as diphenylmethane-4,4'-diisocyanate, also known as methylene-bis(4-phenylisocyanate) or "MDI", 2,4-tolylene diisocyanate, methylene-bis(4-cyclohexylisocyanate), isophorone diisocyanate, tetramethylene-p-xylylene diisocyanate, and the like. MDI is preferred.

Chain extenders used in producing the hard segment of the fibres preferably include one or more of ethylenediamine (EDA), 1,3-propylenediamine, 1,4-cyclohexanediamine, hydrogenated m-phenylenediamine (HPMD), 2-methylpentamethylene diamine (MPMD) and 1,2-propylene diamine. More preferably, the chain extender is one or more of ethylenediamine, 1,3-

propylenediamine, and 1,4-cyclohexanediamine, optionally mixed with HPMD, MPMD and/or 1,2-propylenediamine.

Spandex fibres with poly(tetramethylene) ethers as the soft segments are marketed by DuPont de Nemours International S.A. under the registered trade mark LYCRA® of DuPont de Nemours and Company.

Spandex fibres are generally mixed with other fibres such as cotton, polyamide, wool, polyester and acrylics and made into yarn which is then made into fabric. The content of spandex fibres is usually in a range from 0.5% by weight of the yarn or fabric up to 50%, more usually from 1 to 30% by weight of the yarn or fabric.

A wide range of garments may contain spandex fibres in the fabric, including active sports wear, intimate apparel, hosiery and a variety of ready to wear casual clothing.

The textiles which are treated with a perfume composition prior to wearing may be yarn which is later made into fabric, or may be fabric in the form of a web or lengths from a web which have not yet been made into garments, or may possibly be garments.

Preferably the treatment with a perfume composition is carried out while treating with other material in a conventional process step, especially a

wet stage in which the yarn or fabric is treated with a finishing agent to improve its hand or appearance.

However, the perfume composition may be included directly into the spandex fibre. A fabric  
5 may be made using the spandex fibre alone. Alternatively, the spandex fibre may be covered or mixed with other fibres and made into a yarn which is then made into fabric.

The materials which may be applied to fabric  
10 in a conventional finishing treatment include resins to confer stiffness, fabric stability or permanent press, fabric softeners, flame retardants, fabric brighteners, anti-snap agents, materials to confer soil or stain resistance and water repellants.

15 Techniques which are conventionally used to apply such materials are padding and exhaustion, both well known in the technology of textile manufacture.

Treatment with a perfume composition in accordance with this invention can be carried out by  
20 including the perfume composition in the liquor used in a process as above.

The amount of perfume deposited on the fabric in a treatment step carried out on fabric will generally be from 0.001% to 1% by weight of the  
25 fabric.

Fragrance materials

We have found that a range of fragrance materials deposit well on, or are retained well on, spandex fibres. Such materials include the following two categories:

5     Category A

hydroxylic materials which are alcohols, phenols or salicylates, with an octanol/water partition coefficient (P) whose common logarithm ( $\log_{10}P$ ) is 2.5 or greater, and a gas chromatographic Kovats  
10     index (as determined on polydimethylsiloxane as non-polar stationary phase) of at least 1050.

Category B

esters, ethers, nitriles, ketones or aldehydes, with an octanol/water partition coefficient (P)  
15     whose common logarithm ( $\log_{10}P$ ) is 2.5 or greater, and a gas chromatographic Kovats index (as determined on polydimethylsiloxane as non-polar stationary phase) of at least 1300.

The octanol-water partition coefficient (or  
20     its common logarithm 'logP') is well known in the literature as an indicator of hydrophobicity and water solubility (see Hansch and Leo, *Chemical Reviews*, **71**, 526-616, (1971); Hansch, Quinlan and Lawrence, *J. Organic Chemistry*, **33**, 347-350 (1968). Where such  
25     values are not available in the literature they may be measured directly, or estimated approximately using

mathematical algorithms. Software providing such estimations are available commercially, for example 'LogP' from Advanced Chemistry Design Inc.

A requirement for  $\log_{10}P$  of 2.5 or more calls  
5 for materials which are somewhat hydrophobic.

Kovats indices are calculated from the retention time in a gas chromatographic measurement referenced to the retention time for alkanes [see Kovats , *Helv.Chim.Acta* **41**, 1915 (1958)]. Indices  
10 based on the use of a non-polar stationary phase have been used in the perfumery industry for some years as a descriptor relating to the molecular size and boiling point of ingredients. A review of Kovats indices in the perfume industry is given by T  
15 Shibamoto in "Capillary Gas Chromatography in Essential Oil Analysis", P Sandra and C Bicchi (editors), *Huethig* (1987), pages 259-274. A common non-polar phase which is suitable is 100% dimethyl polysiloxane, as supplied for example under a variety  
20 of tradenames such as HP-1 (Hewlett-Packard), CP Sil 5 CB (Chrompack), OV-1 (Ohio Valley) and Rtx-1 (Restek).

Materials of low Kovats index tend to be volatile and are not retained well on many fibres.

We have found that when perfumery materials  
25 have partition coefficient as above and a relatively high value of Kovats index, deposition and retention

on spandex tends to be greater than on other fibres. Preferably therefore, the perfume composition contains at least 50 wt %, better at least 70 or 80 wt % of materials from the categories above.

5                   We have found that there is a particularly high enhancement of deposition and retention on spandex, compared to other fibres, with materials within the above categories and having a Kovats index of not more than 1600. These sub-sets of categories A and B may be termed categories A' and B'. Preferably  
10                   therefore, the perfume composition contains at least 10 wt%, better at least 20 wt% or 25 wt% of such materials. In some preferred perfumes the amount of fragrance materials from categories A' and B' is at  
15                   least 40 wt% in total.

                  Such fragrance materials are of mid-range volatility (i.e. intermediate between the volatile perfume materials used as "top-notes" and the materials of low volatility which are customarily used  
20                   as "base notes" in perfumes). These materials of mid-range volatility are often not perceptible on other fabrics such as cotton, polyamide and polyester after washing and drying.

                  Category A includes alcohols of general  
25                   formula ROH where the hydroxyl group may be primary, secondary or tertiary, and the R group is an alkyl or



alkenyl group, optionally branched or substituted,  
cyclic or acyclic, such that ROH has partition  
coefficient and Kovats properties as defined above.  
Alcohols of Kovats index 1050 to 1600 are typically  
5 monofunctional alkyl or arylalkyl alcohols with  
molecular weight falling within the range 150 to 230.

Category A also includes phenols of general  
formula ArOH, where the Ar group denotes a benzene  
ring which may be substituted with one or more alkyl  
10 or alkenyl groups, or with an ester grouping  $-CO_2A$ ,  
where A is a hydrocarbon radical, in which case the  
compound is a salicylate. ArOH has partition  
coefficient and Kovats index as defined above.  
Typically, such phenols with Kovats index 1050 to 1600  
15 are monohydroxylic phenols with molecular weight  
falling within the range 150 to 210.

A sub-set of fragrance materials which are  
particularly preferred are those with a partition  
coefficient of 1000 or more, i.e.  $\log_{10}P$  of 3 or more,  
20 and a Kovats parameter of 1100 up to 1600.

Some examples of hydroxylic ingredients  
which fulfil the above criteria for category A' are  
listed as a table below. Materials which are in the  
particularly preferred sub-set are marked with an  
25 asterisk. Semitrivial names are those used in standard  
texts known within the perfume industry, particularly:

*Common Fragrance and Flavor Materials* by Bauer, Garbe and Surburg, VCH Publ., 2nd edition (1990), and *Perfume and Flavour Materials*, Steffen Arctander, published in two volumes by the author (1969).

5	Examples of fragrance materials in category A'
	1-(2'-tert-butylcyclohexyloxy)-butan-2-ol*
	3-methyl-5-(2',2',3'-trimethylcyclopent-3-enyl)-pentan-2-ol*
	4-methyl-3-decen-5-ol*
10	amyl salicylate*
	2-ethyl-4(2',2',3'-trimethylcyclopent-3'-enyl)but-2-enol* (Bangalol, TM)
	borneol*
	carvacrol*
15	citronellol*
	9-decenol*
	dihydroeugenol*
	dihydrolinalol*
	dihydromyrcenol
20	dihydroterpineol *
	eugenol
	geraniol*
	hydroxycitronellal*
	isoamyl salicylate*
25	isobutyl salicylate*
	isoeugenol*
	linalol
	menthol*
	nerolidol*

	nerol*
	para tert-butyl cyclohexanol*
	phenoxanol*
	terpineol
5	tetrahydrogeraniol*
	tetrahydrolinalol
	tetrahydromyrcenol
	thymol*
	2-methoxy-4-methylphenol (Ultravanil, TM)
10	(4-isopropylcyclohexyl)-methanol*

Some examples of fragrance materials which are in category A but which have Kovats index above 1600 (so as to fall outside category A') are:

benzyl salicylate  
 15 cyclohexyl salicylate  
 hexyl salicylate  
 patchouli alcohol  
 farnesol

Category B is esters, ketones, aldehydes,  
 20 nitriles or ethers which have an octanol-water partition coefficient whose common logarithm ( $\log_{10}P$ ) is at least 2.5, and a Kovats index of at least 1300 (non-polar phase).

Ingredients of Category B are of general  
 25 formula  $RX$ , where  $X$  may be in a primary, secondary or tertiary position, and is one of the following groups:

-COA, -OA, -CO<sub>2</sub>A, -CN or -CHO. The groups R and A are hydrocarbon residues, cyclic or non-cyclic and optionally substituted. In some forms of this invention, category B excludes any material with a free hydroxy group, so that where a hydroxyl group is present, the material should be considered only for Category A membership. Typically, the materials of Category B with Kovats index not exceeding 1600 (which may be called category B') are monofunctional compounds with molecular weights in the range 160 to 230.

A sub-set of particularly preferred fragrance materials within category B' is those with a Kovats parameter falling within the range 1350 up to 1600, and possessing a molecular structure containing a ring, such as phenyl or cycloalkyl.

A number of fragrance materials which fulfil the above criteria for category B' are listed in the table below. Materials which are in the particularly preferred sub-set are marked with an asterisk.

	Examples of fragrance materials in category B'
	1-methyl-4-(4-methyl-3-pentenyl)-3-cyclohexene-1-carbaldehyde*
	1-(5',5'-dimethylcyclohexenyl)-pent-en-1-one*
25	2-heptyl cyclopentanone*

	2-methyl-3-(4'-tert-butylphenyl)propanal	
	2-methylundecanal	
	2-undecenal	
	2,2-dimethyl-3-(4'-ethylphenyl)-propanal	
5	3-(4'-isopropylphenyl)-2-methylpropanal	
	4-methyl-4-phenylpent-2-yl acetate*	
	allyl cyclohexyl propionate*	
	allyl cyclohexyloxyacetate*	
	amyl benzoate*	
10	methyl ethyl ketone trimers	(Azarbre, TM)
	benzophenone*	
	3-(4'-tert-butylphenyl)-propanal	(Bourgeonal, TM)
	caryophyllene*	
	cis-jasmone*	
15	citral diethyl acetal	
	citronellal diethyl acetal	
	citronellyl acetate	
	phenylethyl butyl ether	(Cressanther, TM)
	damascone, alpha-*	
20	damascone, beta-*	
	damascone, delta-*	
	decalactone, gamma-*	
	dihydro isojasmonate*	
	dihydrojasmone*	
25	dihydroterpinyl acetate	
	dimethyl anthranilate*	
	diphenyl oxide*	
	diphenylmethane*	
	dodecanal	
30	dodecen-2-al	
	dodecane nitrile	

	1-ethoxy-1-phenoxyethane	(Efetaal, TM)
	3-(1'-ethoxyethoxy)-3,7-dimethylocta-1,6-diene	(Elintaal Forte (TM))
	4-(4'-methylpent-3'-enyl)-cyclohex-3-enal	(Empetaal, TM)
5	ethyl tricyclo[5.2.1.0~2,6~]decane-2-carboxylate*	
	1-(7-isopropyl-5-methylbicyclo[2.2.2]oct-5-en-2-yl)-1-ethanone*	(Felvinone, TM)
	allyl tricyclodecenyl ether*	(Fleuroxene, TM)
	tricyclodecenyl propanoate*	(Florocyclene, TM)
10	gamma-undecalactone*	
	n-methyl-n-phenyl-2-methylbutanamide*	(Gardamide, TM)
	tricyclodecenyl isobutyrate*	(Gardocyclene, TM)
	geranyl acetate	
	hexyl benzoate*	
15	ionone alpha*	
	ionone beta*	
	isobutyl cinnamate*	
	isobutyl quinoline*	
	isoeugenyl acetate*	
20	2,2,7,7-tetramethyltricycloundecan-5-one*	(Isolongifolanone, TM)
	tricyclodecenyl acetate*	(Jasmacyclene, TM)
	2-hexylcyclopentanone	(Jasmatone, TM)
	4-acetoxy-3-pentyltetrahydropyran*	(Jasmopyrane, TM)
25	ethyl 2-hexylacetoacetate	(Jessate, TM)
	8-isopropyl-6-methylbicyclo[2.2.2]oct-5-ene-2-carbaldehyde	(Maceal, TM)
	methyl 4-isopropyl-1-methylbicyclo[2.2.2]oct-5-ene-2-carboxylate*	
30	methyl cinnamate	
	alpha iso methyl ionone*	
	methyl naphthyl ketone*	

	nerolin	
	nonalactone gamma	
	nopyl acetate*	
	para tert-butyl cyclohexyl acetate	
5	4-isopropyl-1-methyl-2-[1'-propenyl]-benzene*	(Pelargene, TM)
	phenoxyethyl isobutyrate*	
	phenylethyl isoamyl ether*	
	phenylethyl isobutyrate*	
10	tricyclodecanyl pivalate*	(Pivacyclene, TM)
	phenylethyl pivalate*	(Pivarose, TM)
	phenylacetaldehyde hexylene glycol acetal*	
	2,4-dimethyl-4-phenyltetrahydrofuran	(Rhubafuran, TM)
	rose acetone*	
15	terpinyl acetate	
	4-isopropyl-1-methyl-2-[1'-propenyl]-benzene	(Verdoracine, TM)
	yara*	
	(4-isopropylcyclohexadienyl)ethyl formate	

20                    Examples of fragrance materials which lie within category B, but have Kovats index above 1600 and so are outside category B' are listed in the following table:

	Within category B but outside category B'
25	amyl cinnamate
	amyl cinnamic aldehyde
	amyl cinnamic aldehyde dimethyl acetal
	cinnamyl cinnamate

5	1,2,3,5,6,7,8,8a,-octathyro-1,2,8,8-tetramethyl-2-acetyl naphthalene (iso E super (TM))
	cyclo-1,13-ethylenedioxytridecan-1,13-dione (ethylene brassylate)
	cyclopentadecanolide (Exaltolide, TM)
	hexyl cinnamic aldehyde
10	1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethylcyclopenta[g]- 2-benzopyran (Galaxolide, TM)
	geranyl phenyl acetate
	6-acetyl-1-isopropyl-2,3,3,5-tetramethylindane (Traseolide, TM)
	1,1,2,4,4,7-hexamethyl-6-acetyl-1,2,3,4- tetrahydronaphthalene (Tonalid, TM)

15                   As indicated above, it is particularly  
preferred to utilise a perfume composition which has  
deodorant properties. Preferably, the perfume is a  
deodorant perfume giving a Malodour Reduction Value of  
at least 0.5, preferably at least 0.9, in the Malodour  
20 Reduction Value test described below and which is an  
adaptation of the test described in EP-A-147191 and  
corresponding US-A-4663068.

The Malodour Reduction Value Test

25                   In this test, the Malodour Reduction Value  
of a deodorant perfume is measured by assessing its  
effectiveness, when applied to fabric, in reducing  
body malodour when the fabric so treated is placed in



contact with the axillae (armpits) of a panel of human subjects, and held there for a standard period of time. From subsequent olfactory evaluation by trained assessors, a Malodour Reduction Value can be  
5 calculated so giving a measure of the effectiveness as a deodorant of the perfume under test.

Stage 1 is preparation of the perfume treated fabric. A test fabric is subjected to a textile finishing which applies perfume to the fabric  
10 at a predetermined percentage of perfume composition, by weight of the cloth. A control fabric is given similar treatment, with or without perfume, depending on the purpose of the test. Depending on the purpose of the test, the fabrics may subsequently be washed  
15 and dried.

The test and control fabrics are cut into 20cm x 20cm squares for testing.

Stage 2 is the carrying out of the test. A team of three Caucasian female assessors of minimum  
20 age 20 years is selected to carry out olfactory evaluation on the basis that each is able to rank correctly the odour levels of the series of standard aqueous solutions of isovaleric acid listed below, and each is able to assign a numerical score,  
25 corresponding to the odour intensity of one of these solutions, to the body malodour of a shirt insert

after has been worn in the axillary region by a male subject for a standard period of time.

A panel of 40 human subjects for use in the test is assembled from Caucasian male subjects of age within the range of from 20 to 55 years. By screening, subjects are chosen who develop axillary body malodour that is not unusually strong and who do not develop a stronger body malodour in one axilla compared with the other. Subjects who develop unusually strong body malodour, for example due to a diet including curry or garlic, are not selected for the panel.

For two weeks before the start of the test, the panel subjects are assigned an unperfumed, non-deodorant soap bar for exclusive use when washing and are denied the use of any other type of deodorant or antiperspirant. At the end of this period, the 40 subjects are randomly divided into two groups of 20.

The "test" and "control" fabric pieces are then tacked into 40 clean cotton or polyester cotton shirts in the underarm region in such a manner that in 20 shirts, the control fabric pieces are attached inside the left underarm region, and the test fabric pieces are attached in the right underarm region. For the remaining 20 shirts, the placing of control and test pieces of fabric is reversed.

The shirts carrying the tacked-in fabric

inserts are then worn by the 40 panel members for a period of 5 hours, during which time each panellist performs his normal work function without unnecessary exercise.

5                   After this five hour period, the shirts are removed and the inserts detached and placed in polyethylene pouches prior to assessment by the trained panel of assessors.

                  The malodour intensity of each fabric insert  
10                   is evaluated by all three assessors who, operating without knowledge of which inserts are "test" and which are "control" and, without knowing the scores assigned by their fellow assessors, sniff each fabric piece and assign to it a score corresponding to the  
15                   strength of the odour on a scale from 0 to 5, with 0 representing no odour and 5 representing very strong odour.

                  Standard aqueous solutions of isovaleric acid which correspond to each of the scores 1, 2, 3, 4  
20                   and 5 are provided for reference to assist the assessors in the malodour evaluation. These are shown below:

SCORE	ODOUR LEVEL	CONCENTRATION OF AQUEOUS ISOVALERIC ACID (ML/L)
0	NO ODOUR	0

1	SLIGHT	0.013
2	DEFINITE	0.053
3	MODERATE	0.22
4	STRONG	0.87
5	VERY STRONG	3.57

The scores recorded by each assessor for each fabric piece are averaged. The average score of the "test" fabric pieces is deducted from the average score of the "untreated" control fabric pieces to give a Malodour Reduction Value.

As a check that the selection of panel subjects is satisfactory for operation of the test, the average score with unperfumed fabric pieces should be between 2.5 and 3.0.

Preferred deodorant perfumes are those which have a Malodour Reduction Value of at least 0.50, or 0.70, or 1.00. The higher the minimum value, the more effective is the perfume as a deodorant as recorded by the assessors in the Malodour Reduction Value Test. It has also been noted that consumers, who are not trained assessors, can detect by self-assessment a noticeable reduction in malodour on soiled fabric such as shirts and underclothes where the Malodour Reduction Value is at least 0.30, so the higher the Malodour Reduction Value above this figure, the more

noticeable is the deodorant effect.

Selection of a combination of fragrance materials to give a deodorant effect is explained in patents such as US-A-430679 referred to earlier.

5 Further systems of selection are given in US-A-5482635 and US-A-5554588 also mentioned above.

Such selections can be carried out using materials with preferred values of partition coefficient and Kovats index as discussed above.

10 US-A-5501805 describes perfume compositions made from a combination of fragrance materials, where the composition is a deodorant perfume yet has a relatively low odour. Such "low-odour" deodorant perfumes may be used in the present invention.

15 When this test is used to assess the deodorant effectiveness of a perfume composition, applied to fabric in accordance with this invention, the test fabric is a blend of 95% by weight cotton and 5% by weight spandex fibres. The control fabric is  
20 100% cotton. The test and control fibres are selected to be similar in other respects, in particular to have the same weight per unit area.

The test fabric is subjected to treatment with a fabric finishing liquor, containing perfume, so  
25 as to apply 0.5% of the perfume, by weight of the fabric. The control fabric is treated similarly, but

without perfume.

The test and control fabrics are not subsequently washed before testing.

However, this test procedure can be operated  
5 in other ways. To demonstrate the higher deposition of perfume on spandex fibres, the control and test fabrics are both treated with the same fabric finishing liquor containing perfume. To isolate the deodorant effect of the perfume, the test and control  
10 fabrics can be the same, but no perfume is present in the liquor used to treat the control fabric.

Example 1

This model experiment demonstrates perfume deposition on spandex fibres. A mixture of perfume  
15 ingredients was prepared and added to an unperfumed, but otherwise conventional, laundry detergent powder, to provide a perfume concentration of 0.5% by weight.

The perfumed powder was used to wash test cloths which had not previously been treated with any  
20 perfume. These were either all cotton, or 95% cotton with 5% spandex. After washing, the cloths were rinsed and then line dried overnight.

The perfume was extracted from the dry cloths with organic solvent, and the content of the  
25 perfume ingredients in the solvent extracts was determined by gas chromatography. If the concentration

of an ingredient extracted from the spandex-containing cloth was greater than from the all-cotton cloth by a factor of 5 to 20, the result was coded as a medium enhancement (M). If the concentration was greater by 20 or more, it was coded high (H) and if less than 5 or not measurable, it was coded (L). The results obtained were as follows:

	Ingredient	K*	logP* *	Enhancement	Category
	Boisambrene Forte	1714	5.5	M	B
10	benzyl acetone	1206	2.0	M	-
	citronellol	1209	3.6	H	A'
	2,6-dimethyl-heptan-2-ol	975	2.9	L	-
	jasmacyclene	1394	2.9	H	B'
15	methyl salicylate	1167	2.3	L	-
	2-phenylethanol	1087	1.4	L	-
	terpinyl acetate	1331	4.0	H	B'
	tetrahydrogeraniol	1180	3.6	H	A'
	tetrahydrolinalol	1083	3.5	H	A'
20	Tonalid	1840	6.4	M	B
	yara	1416	3.2	H	B'

\* Measured on OV-1 polydimethylsiloxane (Ohio Valley) as stationary phase using capillary gc

\*\* Measured or estimated using 'logP' software from ACD  
25 Inc.

Example 2

Cloth composed of 90% cotton, 10% spandex was treated in a fabric finishing step, using conventional equipment. Other cloth, consisting of cotton only, was treated in the same way. In both cases, the fabric finishing treatment was carried out for a period of 20 minutes, using an MCS jet machine (Urgano, Italy). The finishing liquors were applied applied at a liquor to cloth ratio of 20:1, at 40°C with a pH of 5.5. These liquors all contained Ceranine HCS (a finishing agent made by Sandoz) at a concentration such that this agent was applied to fabric at 1% by weight of the fabric.

The treatment liquors also contained perfume at varying concentrations, so that this was applied to the fabric at concentrations of 0.01%, 0.1% and 1% by weight of the fabric. Liquor without perfume was used to provide a control.

The perfume was used, designated "perfume U" contained 33.5% (by weight of the perfume composition) of fragrance materials in category A above, all of which had Kovats indices of 1050 to 1600 and therefore all fell within category A'. The perfume also contained 41.1% (by weight of the perfume composition) of materials in category B. These were made up from 26.7% with Kovats index over 1600 and 14.4% with



Kovats index of 1300 to 1600, so as to fall within category B'.

The cloths were then washed repeatedly, using a commercial detergent powder which included a different perfume. The washes were carried out using a Miele washing machine on its "Quickwash" programme at 40°C. 110gm of detergent powder was used for each wash. The fabric was rinsed three times after each wash and tumble dried.

The dried cloths were examined by a panel of expert assessors of perfume intensity. This was to determine the intensity of perfume on the fabric, but not its deodorant property. The results obtained were as follows:

	Odour Evaluation Scores						
	100% cotton			90% cotton and 10% spandex			
15	Washes:	1	3	5	1	3	5
	Perfume U						
	0%	2.0	2.4	2.8	4.0	4.4	4.6
	1.0%	6.4	3.6	<3	16.0	14.0	12.8
	0.1%	3.6	<3	<3	10.4	9.7	9.0
20	0.01%	3.2	<3	<3	8.8	8.0	7.2

It can be seen that the cloths which were not perfumed in the finishing treatment took up

perfume in the first wash and this perfume built up slowly in subsequent washes. The quantity of perfume taken up was greater on the cloth which included spandex fibres.

5                   The cloths containing spandex fibres which were perfumed during the finishing treatment had a much higher level of perfume on them after one wash than the 100% cotton cloths. Even after five washes, the intensity of perfume on them exceeded the intensity  
10 of perfume on the 100% cotton cloths after one wash, and on the cloths which had not been perfumed prior to the first wash. Thus the spandex fibres were providing enhanced retention of perfume as well as enhanced deposition.

15                   The 100% cotton cloths which had been perfumed during the finishing treatment were assessed again after 3 and 5 washes. The results showed that the level of intensity of the perfume was less than that observed after 1 wash but also showed that the  
20 olfactive differences between perfume U used in the finishing treatment and the perfume present in the washing powder was confusing the panellists.

### Example 3

                  Two deodorant perfumes were used in  
25 treatment of cloths by a finishing process as in Example 2.

Perfume L contained materials in the above categories as follows:

- Category A: 30 wt% (all with Kovats index above 1600)
- 5 Category B: 68.5 wt% (13% with Kovats index 1300-1600 and therefore within category B', and 55.5 wt% with Kovats index above 1600).

Perfume M contained materials in the above categories  
10 as follows:

- Category A: 24.9 wt% (16.3 wt% with Kovats index 1050-1600 and therefore within category A', and 8.6 wt% with Kovats index above 1600)
- 15 Category B: 55.3 wt% (8.6 wt% with Kovats index 1300-1600 and therefore within category B', and 46.7 wt% with Kovats index above 1600).

The test cloths were: 100% cotton, 90%  
20 cotton with 10% spandex, 95% cotton with 5% spandex, 100% nylon and 82% nylon with 18% spandex. Perfume was used at a concentration of 0.5% based on the weight of the fabric. The treated cloths were tested for Malodour reduction in the test described earlier. The  
25 control cloths were 100% cotton, which had been subjected to the same finishing treatment, but without

perfume in that finishing treatment. The results are set out in the following tables, which show substantial enhancements of malodour inhibition when fabrics containing spandex fibres were used.

5 Test 1:

Fabric		perfume M (% by weight of fabric)	Malodour score	Malodour reduction	Malodour reduction as % of control
other fibre	spandex				
10 90% cotton	10%	0.5%	1.19	1.46	55%
100% cotton	0	0.5%	1.92	0.73	27.4%
82% nylon	18%	0.5%	1.00	1.65	62.1%
15 100% nylon	0	0.5%	1.97	0.68	25.5%
100% cotton (control)	0	0	2.65		

Notes: % malodour reduction calculated as 100%

20  $x(\text{control score} - \text{sample score}) / \text{control score}$

Statistical calculation showed that a difference in malodour reduction of 6.9% was significant at 95% level of confidence.

## Test 2:

	Fabric		perfume (% by weight of fabric)	Malodour score	Malodour reduction	Malodour reduction as % of control
	other fibre	spandex				
5	95% cotton	5%	0.5% L	1.15	1.22	51.5%
	95% cotton	5%	0.5% M	1.29	1.08	45.6%
10	100% cotton (control)	0	0	2.37		

Note: Statistical calculation showed that a difference in malodour reduction of 6.3% was significant at 95% level of confidence.

15 In test 1, malodour scores on 100% cotton fabric, with and without perfume, demonstrate a malodour reduction value of 0.73 attributable to the perfume. A similar malodour reduction value was observed when the test cloth was 100% nylon.

20 When spandex fibre was incorporated, the malodour reduction increased greatly, showing that increased deposition of perfume on spandex fibres compared with other fibres also provides an increased deodorant efficiency.

25 In test 2, similar high values of malodour reduction were obtained when either perfume L or

perfume M was used, in test fabrics with 5% by weight spandex fibres.

CLAIMS

1. A method of treating textile which is yarn or unworn fabric containing spandex fibres, comprising contacting the textile with a perfume composition  
5 which is a mixture of fragrance materials, so that fragrance materials are deposited on the textile.
2. A method according to claim 1 wherein the textile contains from 0.5 wt % to 50 wt % spandex fibres and the amount of fragrance materials deposited  
10 on the textile is from 0.001% to 1% by weight of the textile.
3. A method of treating a spandex fibre comprising contacting the fibre with a perfume composition which is a mixture of fragrance materials, so that fragrance  
15 materials are deposited on the spandex fibre.
4. A method according to claim 1, claim 2 or claim 3 wherein the perfume composition contains at least 50%, by weight of the perfume composition, of fragrance materials selected from:  
20 Category A) hydroxylic materials which are alcohols, phenols or salicylates, with an octanol/water partition coefficient (P) whose common logarithm ( $\log_{10}P$ ) is 2.5 or greater, and a gas chromatographic Kovats index (as  
25 determined on polydimethylsiloxane as non-polar

stationary phase) of at least 1050, and

Category B) esters, ethers, nitriles, ketones or aldehydes, with an octanol/water partition coefficient (P) whose common logarithm ( $\log_{10}P$ ) is 2.5 or greater, and a gas chromatographic Kovats index (as determined with polydimethylsiloxane as non-polar stationary phase) of at least 1300.

5. A method according to claim 4 wherein the perfume composition contains at least 10%, by weight of the perfume composition, of fragrance materials selected from:

Category A') hydroxylic materials which are alcohols, phenols or salicylates, with an octanol/water partition coefficient (P) whose common logarithm ( $\log_{10}P$ ) is 2.5 or greater, and a gas chromatographic Kovats index (as determined on polydimethylsiloxane as non-polar stationary phase) lying within the range 1050 to 1600, and

Category B') esters, ethers, nitriles, ketones or aldehydes, with an octanol/water partition coefficient (P) whose common logarithm ( $\log_{10}P$ ) is 2.5 or greater, and a gas chromatographic



Kovats index (as determined on polydimethylsiloxane as non-polar stationary phase) lying within the range 1300 to 1600.

6. A method according to claim 4 wherein the  
5 perfume composition contains at least 70%, by weight of the perfume composition, of fragrance materials selected from categories A and B, which materials include at least 25% by weight of the perfume composition, of fragrance materials from the  
10 categories A' and B' defined in claim 4.
7. A method according to claim 6 wherein the perfume composition contains at least 80%, by weight of the perfume composition, of fragrance materials selected from categories A and B, which materials  
15 include at least 40%, by weight of the perfume composition, from categories A' and B'.
8. The method of claim 1, wherein the textile is contacted with the perfume composition in a fabric finishing step.
- 20 9. A textile which is yarn or unworn fabric and which contains spandex fibres, having fragrance materials deposited on the textile.
10. A textile according to claim 9 which contains from 0.5 to 50 wt % spandex fibres and the amount of  
25 fragrance materials deposited thereon is from 0.001% to 1% by weight of the textile.

11. A textile according to claim 9 or claim 10 which is yarn, a fabric web or length thereof, or an unworn garment.
12. A textile which comprises spandex and other  
5 fibres and which has fragrance materials preferentially deposited on the spandex fibres.
13. A spandex fibre having fragrance materials deposited on the spandex fibre.
14. Use of a perfume composition which is a mixture  
10 of fragrance materials in the treatment of yarn or unworn fabrics containing spandex and other fibres, to deposit fragrance materials on the spandex fibres.



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Int. application No. : PCT/GB98/03056  
Our Reference : 85/DP01L49/WO  
Owner/Applicant : E.I. DUPONT DE NEMOURS AND COMPANY  
Subject : METHOD FOR TEXTILE TREATMENT FOR  
SPANDEX CONTAINING FABRICS

file: LP-4285  
(PCT)

This is Applicant's response to the Written Opinion mailed July 02, 1999 in the above-referenced Application. A (extended) five month period was set for response.

It is herewith submitted an amended set of claims 1 to 11 which is to replace the set of claims 1 to 14 as hitherto on file.

The correlation of the amended claims and the claims as hitherto on file can be gathered from the following table:

amended claim	1	2	3	4	5	6
claim(s) as hitherto on file	1+4	2	3+4	5	6	7

amended claim	7	8	9	10	11	
claim(s) as hitherto on file	8	9+4	10	12+4	13+4	

As can be gathered from the above table, subject matter of claim 4 as hitherto on file was incorporated into amended claims 1, 8, 10 and 11, claims 11 and 14 as hitherto on file were cancelled.

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Incorporation of subject matter of claim 4 as hitherto on file into the amended claims 8, 10 and 11 is allowable as these amendments do not go beyond the disclosure in the international application as filed.

For the convenience of the international preliminary examining authority, Applicant has organised the response by making reference to the numbered paragraphs as they appear in the Written Opinion.

Concerning item V, 1.3:

Claim 14 as hitherto on file was cancelled

Concerning item V, 2.1 to 2.3:

By incorporation of subject matter of claim 4 as hitherto on file, the subject matter claimed by the independent claims is limited to such methods, textiles and fibres which all use or comprise fragrance compositions as defined in claim 4 as hitherto on file.

The disclosure of claim 4 as hitherto on file defines fragrance materials (categories A and B) by the possible classes of compounds and in addition by two physical properties (octanol/water partition coefficient, gas chromatographic Kovats index) which values must lie above specific limits. The determination of the physical values as defined in claim 4 as hitherto on file can be achieved by standard techniques as is e.g. disclosed on page 8, lines 19 to page 9, line 21.

Moreover, numerous examples for applicable components falling within category A (resp. sub-category A') or category B (resp. sub-category B') are disclosed in the specification (page 12, line 5 to page 13, line 19; page 13, line 14 to 18; page 14, line 21 to page 17, line 19; page 17, line 24 to page 18, line 14).

Therefore, a person skilled in the art is able to carry out the present invention without undue burden within substantially the whole scope of the amended claims. This is true not only for the substance claims but also for the method claims.



Concerning item V, 2.6 to 3.2

First it should be noted that, as regards novelty (Art. 33(2) PCT) of the subject matter as claimed by the originally filed claims, the preliminary international examining authority did not cite or discuss any specific document. However, according to Rule 64.1(a) PCT for the purposes of Art. 33(2) and (3) PCT, everything made available to the public anywhere in the world by means of written disclosure ... shall be considered prior art ....

But even if one puts the prerequisites of Rule 64(1) PCT aside, no hint can be found (in writing or as "general knowledge") that the features of the independent claims as now on file are known in the art. This question as well as the question of inventive step will be discussed in the following with respect to the documents as cited in the International Search Report which all are considered to be not of particular relevance (category A).

GB 1 596 792 A discloses a fabric conditioning composition comprising an effective amount of a fabric softener and a deodorising amount of a deodorant perfume (claim 1). The deodorant perfume according to GB 1 596 792 A has to comprise materials which depress the partial vapour pressure of morpholine. As according to the present invention, a fabric softener is not part of the invention and the claimed fragrance materials are different from that as disclosed in GB 1 596 792 A the subject matter of the present invention is novel. Moreover, as GB 1 596 792 A gives no hint to use a quite specific selection of perfume materials for textiles containing spandex fibres or for spandex fibres as is disclosed in the present application for achieving the inventive advantages, subject matter of the present invention has to be considered not only as novel but also as inventive in respect to GB 1 596 792 A.

DE 296 00 628 U1 describes a vaporisation liquid for steam irons, which may comprise a perfume compound. Therefore, this document discloses one of the numerous methods by which a fragrance material can be applied to a textile. However, no hint can be found to use fragrance materials as are disclosed in the present application. Therefore the subject matter of the present invention is novel and inventive also in respect to DE 296 00 628 U1.

DE 40 11 928 A1 discloses a textile with an impregnation consisting of fragrance materials together with additional means to make said fragrance materials sustainable and resistant to cleaning agents. Said additional means are either waxes or wax-like components or substances in which the fragrance materials have a good solubility. Finally, in DE 40 11 928 A1 disclosed are micro capsules as additional means. From that it becomes evident that DE 40 11 928 A1 has a quite different teaching as compared to the present invention and, therefore, the subject matter of the present application is novel and inventive in respect to DE 40 11 928 A1, too.



DE 42 42 327 A1 which is according to the preliminary international examining authority – among others - reflecting the closest prior art, discloses a method for finishing textile materials by applying micro capsules into the knit fabric. One of the possible contents of the micro capsules are perfume materials. However, no hint can be found to chose one of the fragrance materials as disclosed in the present application to achieve the inventive advantages of the present invention. Therefore, also DE 42 42 327 A1 does not disclose a subject matter which can destroy novelty and inventive step of the subject matter as disclosed in the present application.

EP 0 581 274 A1 discloses a method comprising the steps of cationising a textile product with a liquid containing a nitrogenous cationic compound and fixing micro-capsules which are made essentially of a polymer compound and which contain a perfume to the textile product. Therefore, same as stated above in respect of DE 42 42 327 A1 is true for the subject matter of EP 0 581 274 A1.

WO 93/14259 discloses a method for applying substances to fibre materials and textile substrates from a liquid phase, in which a supercritical fluid or a liquefied gas or gas mixture is used as a fluid. No hint can be found in the description of WO 93/14259 to use fragrance materials as are used in the present invention.

JP 1272875 A concerns a fragrant non-woven fabric on which micro capsules are adhered; JP 6146112 A concerns a two layer spandex fibre in which one layer comprises a Ag based inorganic antibacterial agent to give the two layer spandex fibre antibacterial properties; finally, EP 0 841 391 A1 discloses a composition comprising a perfume and an amino-functional polymer. Therefore, also these three last documents do neither disclose the subject matter of the present application nor do they render the use of the fragrance composition according to the present invention obvious.

To sum up, the subject matter of the present application is patentable as neither the "general background knowledge " – though formally irrelevant – nor the above discussed documents disclose the subject matter as is disclosed in the present application, and, further, neither alone nor in combination the documents forming the state of the art make it obvious to use the fragrance compositions as are used in the subject matter of the present application to achieve the inventive advantages.




Concerning item VII, 1

The request of the Preliminary International Examining Authority that the documents cited in the search report should be acknowledged in the description is respectfully traversed. As becomes evident from the discussion above the documents as cited in the international search report do not, in the opinion of the applicant, reflect state of the art being closer to the subject matter of the present application as compared to the state of the art which is disclosed in the documents already discussed in the present application (page 2, lines 22 to 25).

Concerning item VIII

The entirety of the claims as now on file is, according to the opinion of the applicant, concise as requested by Art. 6 PCT. Both the method claims and the substance claims are linked by a common inventive concept. This becomes evident by the fact that all independent claims comprise at least one common inventive feature.



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Encl.:  
Amended set of claims 1 to 11 (in triplicate)